

Fluxana, Deutschland - Zeolithe FLX-CRM 104

Veranstalter: FluXana GmbH & Co. KG, Borschelstr. 3, 47551 Bedburg-Hau

Ringversuchsmaterial: FLX-CRM 104 (Zeolith)

RV geschlossen: 2010 – 11

Literatur: Zertifikat Ringversuchsausrichter (Laborcode CRB = Lab No. 7)

Hauptelemente [MA%]

	CRB	RV	1sRV	Z-Score
Na ₂ O	19,64	20,06	0,48	0,9
Al ₂ O ₃	33,92	33,74	0,62	0,3
SiO ₂	46,15	45,98	0,5	0,3
K ₂ O	0,121	0,075	0,06	0,8
CaO	0,07	0,063	0,03	0,2
Fe ₂ O ₃ tot	0,02	0,014	0,01	0,5
L.O.I.*	21,58	22,64	1,14	0,9

Legende

CRB: Ergebnisse CRB – **RV:** Ergebnisse Ringversuch -- **1s-RV:** Standardabweichung Ringversuch

Z-Score: Differenz des Messwertes vom Mittelwert des Ringversuchs -- * Wert nicht zertifiziert

Certificate of Analysis

FLX-CRM 104

Reference Material Information

Type: Zeolite (adsorption material)
Form and Size: Granulate, as-produced, 50g
Manufactured by: Chemiewerk Bad Köstritz CWK GmbH, Germany
Packaged and tested by: FLUXANA GmbH & Co.KG, Germany
Certified by: FLUXANA GmbH & Co.KG, Germany

Certified values and their uncertainties

Percentage element by weight

Constituent	Al ₂ O ₃	CaO	Fe ₂ O ₃	K ₂ O	Na ₂ O	SiO ₂	L.O.I
Value ¹	33.74	0.063	0.014	0.075	20.06	45.98	22.64
Uncertainty ²	0.33	0.019	0.003	0.030	0.26	0.27	0.69

Notes: all values (except LOI) apply after ignition at 950°C for 1 hour.

Definitions

- ¹ The above values are the present best estimates of the true content for each component. Each value is a panel consensus, based on the averaged results of an inter laboratory testing program, detailed in values obtained by individual laboratories or methods.
- ² The uncertainty values are coming from the half width confidence interval C(95%). It is equal to $C(95\%) = (t \times s) / \sqrt{n}$ where t is the appropriate Student's value, n the number of acceptable mean values and s the standard deviation.

Certified by

Fluxana GmbH & Co.KG



on 01th November 2010

Dr. Rainer Schramm

Description of the CRM

This reference material is an industrial product and was taken directly from the production stream. The complete batch was sealed into 50g bottles. This material is normally used for the production of molecular sieves.

Intended use

Calibration and control sample for x-ray fluorescence (xrf) analysis.

Instructions for the correct use of the CRM

This material has to be ignited for minimum 1 hour at 950°C prior use. The ignition process must result in a constant weight. The ignited material must be stored in a desiccator not longer than 24h, then reignition might be necessary. The minimum sample quantity for analysis should be 0.5g. The material is moisture sensitive.

For XRF use, ignited samples should be prepared as a fused bead, using e.g. 1 part sample + 8 parts Lithium tetraborate, prepared on an automated fusion machine, and otherwise in accordance with EN ISO 12677:2009.

Hazardous situation

Not hazardous according 67/548/EWG and 1999/45/EG.

Level of homogeneity

The batch was checked for uniformity using a wavelength-dispersive XRF unit, and a test method in conformance with ISO 29581-2:2010.

Using the data from each sample, standard deviation values were derived for each element as an indicator of any non-homogeneity (as determined for the specific sample size taken by the spectrometer).

Traceability

The analytical work performed to assess this material has been carried out by competent, laboratories, from raw material industry. All of the results derived as part of this testing program have traceability to NIST and other national standards, as part of the analytical calibration or process control.

Values obtained by individual laboratories or methods

Lab No.	Al2O3	CaO	Fe2O3	K2O	Na2O	SiO2	L.O.I
1	34,28	0,12	0,01	0,05	20,04	45,59	22,74
2	34,23	0,01	0,01	0,03	20,47	46,57	22,84
3	33,12	0,06	0,03	0,08	20,59	45,68	23,14
4	33,81	0,02	0,02	0,13	19,83	46,26	23,76
5	33,38	0,07	0,02		19,82	45,75	22,50
7	33,92	0,07	0,02	0,12	19,61	46,15	21,58
8	33,42	0,05	0,01	0,02	20,26	46,00	19,37 ¹
9	33,45	0,04	0,03	0,17	20,18	46,01	22,74
10	33,07	0,02		0,12	21,14 ¹	45,36	23,71
11		0,07	0,01	0,05	19,45		
11	35,55 ¹	0,14 ¹	0,02	0,06		44,70 ¹	
12	33,98	0,08	0,02	0,04	19,15	46,66	22,82
13	32,90	0,05	0,01	0,06	20,31	46,48	23,58
14	33,79	0,05	0,01	0,01	20,01	46,14	
14	33,63		0,01	0,02	19,90	46,34	
15	33,63	0,09		0,06	20,43	45,74	22,53
16	33,73	0,08	0,01	0,20 ¹	19,85	46,26	23,00
Mean	33,74	0,06	0,01	0,08	20,06	45,98	22,64
Stddev	0,62	0,03	0,01	0,06	0,48	0,5	1,14
C (95%)	0.33	0.019	0.003	0.030	0.26	0.27	0.69

¹These results show a z-score > 2 however were not excluded.

Methods used

The method most used is x-ray fluorescence analysis with fused bead as sample preparation (EN ISO 12677:2009). This was performed by laboratory 2,3,4,5,6,7,8,9,10,12,13,14,15,16.

Laboratory 1,11 and 14 used ICP method with digestion.

Laboratory 11 used x-ray fluorescence analysis with pressed powder preparation.

Loss on ignition (L.O.I) was determined by gravimetry 1h at 950°C (EN 196-2).

Participating Laboratories

Bachema AG	Switzerland
CeramTec AG	Germany
CRB Analyse Service GmbH	Germany
ESAB	Sweden
FGK Forschungsinstitut fuer Anorganische Werkstoffe-Glas/Keramik- GmbH	Germany
FH Nürnberg FB Werkstofftechnik	Germany
FLUXANA GmbH & Co.KG	Germany
GEKA mbH	Germany
Grothe Rohstoffe GmbH & Co KG	Germany
Holcim (Schweiz) AG	Switzerland
HuK Umweltlabor GmbH	Germany
LERM	France
LSI - Laboratory Services International	Netherlands
terrachem GmbH	Germany
VDZ	Germany
WESSLING Laboratorien GmbH Umweltanalytik	Germany

Further information

This Reference Material has been produced and certified, wherever possible, in accordance with the requirements of ISO Guide 34-2009, ISO Guide 31-2000 and ISO Guide 35-2006, taking into account the requirements of the ISO Guide to the Expression of Uncertainty in Measurement (GUM).

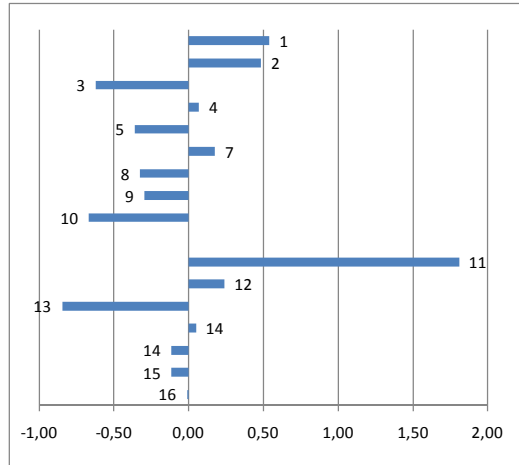
This certification is applicable to the whole of the sample.

As-supplied, this material will not remain stable indefinitely. The matrix will be affected by contact with the atmosphere, and in particular it will absorb moisture. However, it continues to be fit for use for an indeterminate period, on the understanding that the sample will be ignited prior to weighing, bead preparation and measurement.

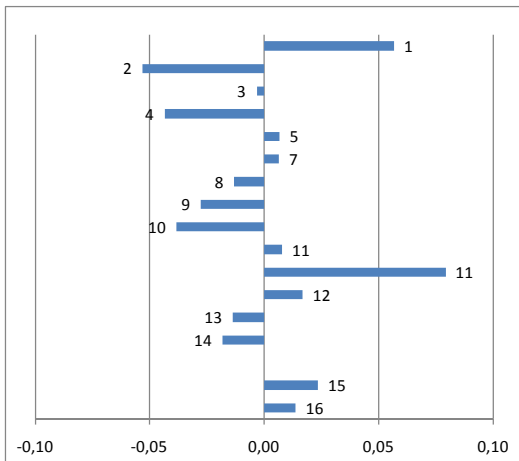
All production records will be retained for a period of 10 years from the date of this certificate. This certification will therefore expire in 31.10.2020, although we reserve the right to make changes as issue revisions, in the intervening period.

The certification, packaging, analysis and storage of this product were supervised by Dr. Rainer Schramm, General Manager, Fluxana GmbH & Co. KG, Bedburg-Hau, Germany.

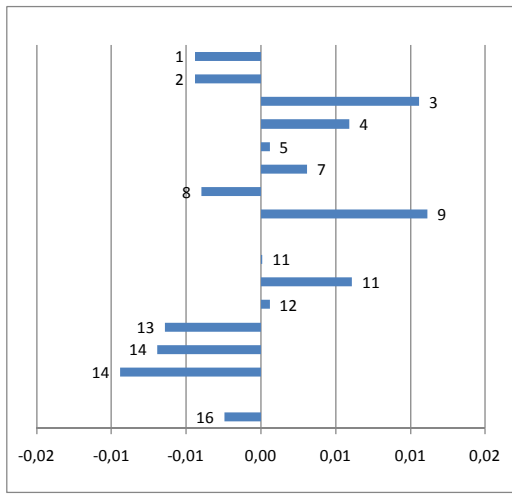
AI2O3							z-score	P(95%) f=16 2,443
Lab. No:	Method	Standard	ISO 17025	% Meas #1	% Meas #2	% Mean		
1	ICP		no	34,28		34,28	0,9	outlier (Grubbs) z-score > 2
2	XRF fusion		yes	34,13	34,32	34,23	0,8	
3	XRF fusion		no	33,190	33,050	33,12	1,0	
4	XRF fusion		no	33,793	33,822	33,81	0,1	
5	XRF fusion		no	33,380	33,380	33,38	0,6	
7	XRF fusion		yes	33,910	33,920	33,92	0,3	
8	XRF fusion		yes	33,36	33,47	33,42	0,5	
9	XRF fusion		yes	33,470	33,420	33,45	0,5	
10	XRF fusion		yes	33,175	32,968	33,07	1,1	
11	ICP		yes	38,8	38,4		2,9	
11	XRF pp		yes	35,6	35,5	35,55	0,4	
12	XRF fusion		no	34,07	33,89	33,98	1,4	
13	XRF fusion	ISO 12677	no	32,9	32,9	32,90	0,1	
14	XRF fusion		no	33,75	33,83	33,79	0,2	
14	ICP		no	33,71	33,54	33,63	0,2	
15	XRF fusion		no	33,630	33,620	33,63	0,2	
16	XRF fusion		no	33,73		33,73	0,0	
						n	16	
						Mean	33,74	
						max	35,55	
						min	32,90	
						s	0,62	
						C(95%)	0,33	



CaO							z-score	P(95%) f=15 2,409
Lab. No:	Method	Standard	ISO 17025	% Meas #1	% Meas #2	% Mean		
1	ICP		no	0,12		0,12	1,6	kleiner Bestimmungsgrenze z-score > 2
2	XRF fusion		yes	0,01	0,01	0,01	1,5	
3	XRF fusion		no	0,050	0,070	0,06	0,1	
4	XRF fusion		no	0,020	0,020	0,02	1,2	
5	XRF fusion		no	0,070	0,070	0,07	0,2	
7	XRF fusion		yes	0,071	0,068	0,07	0,2	
8	XRF fusion		yes	0,05	0,05	0,05	0,4	
9	ICP		yes	0,032	0,039	0,04	0,8	
10	XRF fusion		yes	0,024	0,026	0,02	1,1	
11	ICP		yes	0,075	0,067	0,07	0,2	
11	XRF pp		yes	0,144	0,141	0,14	2,3	
12	XRF fusion		no	0,078	0,082	0,08	0,5	
13	XRF fusion	ISO 12677	no	0,050	0,049	0,05	0,4	
14	XRF fusion		no	0,044	0,046	0,05	0,5	
14	ICP							
15	XRF fusion		no	0,106	0,067	0,09	0,7	
16	XRF fusion		no	0,077		0,08	0,4	
						n	16	
						Mean	0,063	
						max	0,14	
						min	0,01	
						s	0,03	
						C(95%)	0,019	

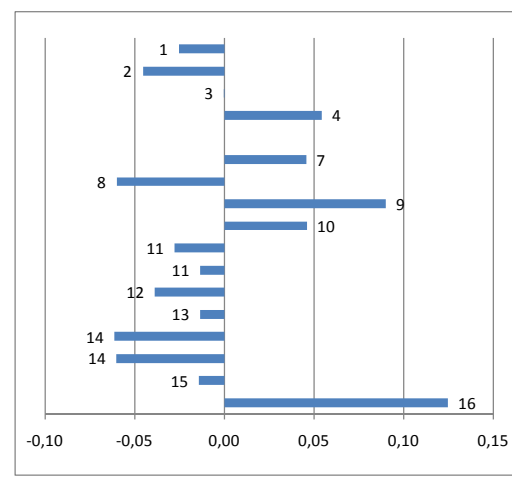


Fe2O3							z-score	P(95%) f=15 2,409
Lab. No:	Method	Standard	ISO 17025	% Meas #1	% Meas #2	% Mean		
1	ICP		no	0,01		0,01	0,7	outlier (Grubbs) kleiner Bestimmungsgrenze kleiner Nachweisgrenze
2	XRF fusion		yes	0,01	0,01	0,01	0,7	
3	XRF fusion		no	0,020	0,030	0,03	1,7	
4	XRF fusion		no	0,018	0,022	0,02	0,9	
5	XRF fusion		no	0,020	0,010	0,02	0,1	
7	XRF fusion		yes	0,020	0,015	0,02	0,5	
8	XRF fusion		yes	0,01	0,01	0,01	0,6	
9	ICP		yes	0,022	0,030	0,03	1,8	
10	XRF fusion		yes	0,065	0,061		0,0	
11	ICP		yes	0,015	0,014	0,01	1,0	
11	XRF pp		yes	0,02	0,021	0,02	0,1	
12	XRF fusion		no	0,017	0,013	0,02	1,0	
13	XRF fusion	ISO 12677	no	0,008	0,008	0,01	1,1	
14	XRF fusion		no	0,007	0,008	0,01	1,5	
14	ICP		no	0,005	0,005	0,01	0,4	
15	XRF fusion		no	<0,009	<0,009			
16	XRF fusion		no	0,012		0,01		
						n	15	
						Mean	0,014	
						max	0,03	
						min	0,01	
						s	0,01	
						C(95%)	0,003	



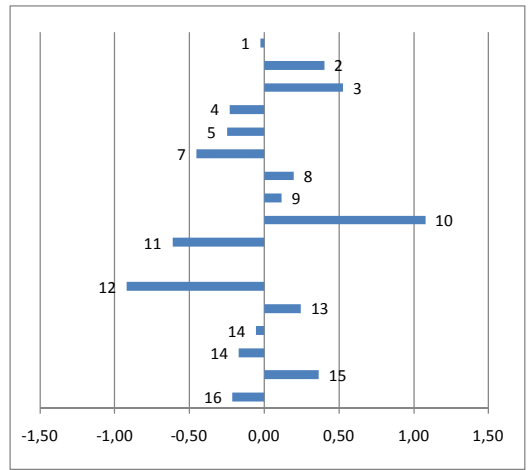
z-score	P(95%) f=15
0,7	2,409
0,7	
1,7	
0,9	
0,1	
0,5	
0,6	
1,8	
0,0	
1,0	
0,1	
1,0	
1,1	
1,5	
0,4	

K2O							z-score	P(95%) f=16 2,443
Lab. No:	Method	Standard	ISO 17025	% Meas #1	% Meas #2	% Mean		
1	ICP		no	0,05		0,05	0,5	outlier (Grubbs) kleiner Bestimmungsgrenze z-score > 2
2	XRF fusion		yes	0,03	0,03	0,03	0,8	
3	XRF fusion		no	0,040	0,110	0,08	0,0	
4	XRF fusion		no	0,130	0,130	0,13	1,0	
5	XRF fusion		no	0,320	0,330		0,8	
7	XRF fusion		yes	0,116	0,126	0,12	1,1	
8	XRF fusion		yes	0,02	0,02	0,02	1,6	
9	ICP		yes	0,183	0,148	0,17	0,8	
10	XRF fusion		yes	0,135	0,108	0,12	1,1	
11	ICP		yes	0,047	0,048	0,05	0,5	
11	XRF pp		yes	0,061	0,063	0,06	0,2	
12	XRF fusion		no	0,034	0,039	0,04	0,7	
13	XRF fusion	ISO 12677	no	0,067	0,057	0,06	0,2	
14	XRF fusion		no	0,013	0,015	0,01	1,1	
14	ICP		no	0,015	0,015	0,02	1,1	
15	XRF fusion		no	0,057	0,065	0,06	0,3	
16	XRF fusion		no	0,2		0,20	2,2	
						n	16	
						Mean	0,075	
						max	0,20	
						min	0,01	
						s	0,06	
						C(95%)	0,030	

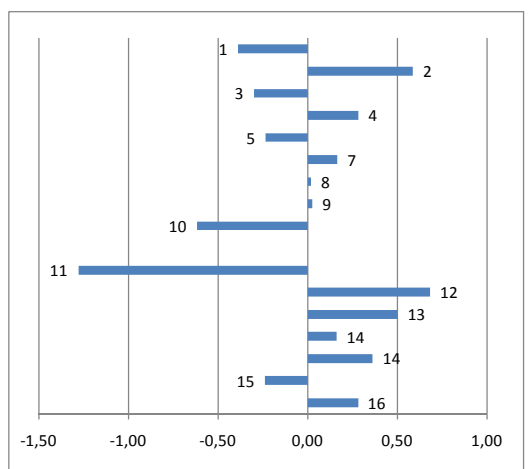


z-score	P(95%) f=16
0,5	2,443
0,8	
0,0	
1,0	
0,8	
1,1	
1,6	
0,8	
1,1	
0,5	
0,2	
0,7	
0,2	
1,1	
1,1	
0,3	
2,2	

Na2O							z-score	P(95%) f=16 2,443
Lab. No:	Method	Standard	ISO 17025	% Meas #1	% Meas #2	% Mean		
1	ICP		no	20,04		20,04	0,1	Kalibrierung nur bis 2% z-score > 2 outlier (Grubbs)
2	XRF fusion		yes	20,45	20,48	20,47	0,8	
3	XRF fusion		no	20,581	20,601	20,59	1,1	
4	XRF fusion		no	19,844	19,826	19,83	0,5	
5	XRF fusion		no	19,770	19,860	19,82	0,5	
7	XRF fusion		yes	19,580	19,640	19,61	0,9	
8	XRF fusion		yes	20,21	20,31	20,26	0,4	
9	XRF fusion		yes	20,110	20,250	20,18	0,2	
10	XRF fusion		yes	20,979	21,304	21,14	2,2	
11	ICP		yes	19,5	19,4	19,45	1,3	
11	XRF pp		yes	17	17,4		1,9	
12	XRF fusion		no	19,18	19,11	19,15	0,5	
13	XRF fusion	ISO 12677	no	20,3	20,4	20,31	0,1	
14	XRF fusion		no	19,96	20,06	20,01	0,4	
14	ICP		no	19,82	19,97	19,90	0,8	
15	XRF fusion		no	20,450	20,410	20,43	0,4	
16	XRF fusion		no	19,85		19,85	0,4	
						n	16	
						Mean	20,06	
						max	21,14	
						min	19,15	
						s	0,48	
						C(95%)	0,26	



SiO2							z-score	P(95%) f=16 2,443
Lab. No:	Method	Standard	ISO 17025	% Meas #1	% Meas #2	% Mean		
1	ICP		no	45,59		45,59	0,8	outlier (Grubbs) z-score > 2
2	XRF fusion		yes	46,42	46,71	46,57	1,2	
3	XRF fusion		no	45,800	45,560	45,68	0,6	
4	XRF fusion		no	46,255	46,265	46,26	0,6	
5	XRF fusion		no	45,670	45,820	45,75	0,5	
7	XRF fusion		yes	46,130	46,160	46,15	0,3	
8	XRF fusion		yes	45,87	46,13	46,00	0,0	
9	XRF fusion		yes	46,040	45,970	46,01	0,1	
10	XRF fusion		yes	45,411	45,314	45,36	1,2	
11	ICP		yes	42,6	39,8		2,6	
11	XRF pp		yes	44,6	44,8	44,70	1,4	
12	XRF fusion		no	46,77	46,55	46,66	1,0	
13	XRF fusion	ISO 12677	no	46,4	46,5	46,48	0,3	
14	XRF fusion		no	46,10	46,18	46,14	0,7	
14	ICP		no	46,45	46,23	46,34	0,5	
15	XRF fusion		no	45,640	45,840	45,74	0,6	
16	XRF fusion		no	46,26		46,26	0,6	
						n	16	
						Mean	45,98	
						max	46,66	
						min	44,70	
						s	0,50	
						C(95%)	0,27	



LOI				%	%	%		
Lab. No:	Method	Standard	ISO 17025	Meas #1	Meas #2	Mean		
1	Gravimetry	EN 196-2	yes	22,74		22,74		
2	Gravimetry		yes	22,82	22,85	22,84		
3	Gravimetry		no	23,155	23,125	23,14		
4	Gravimetry		no	23,750	23,760	23,76		
5	Gravimetry		no	22,500	22,500	22,50		
7	Gravimetry		yes	22,570	20,580	21,58		
8	Gravimetry		yes	19,31	19,42	19,37		
9	Gravimetry		no	22,910	22,560	22,74		
10	Gravimetry			23,730	23,690	23,71		
11	Gravimetry							
11	Gravimetry							
12	Gravimetry		no	22,84	22,8	22,82		
13	Gravimetry		no	23,6	23,6	23,58		
14	Gravimetry		no	17,10	17,25			
14	Gravimetry							
15	Gravimetry		no	22,520	22,530	22,53		
16	Gravimetry		no	23		23,00		
						n	13	
						Mean	22,64	
						max	23,76	
						min	19,37	
						s	1,14	
						C(95%)	0,69	

	z-score	P(95%) f=13
		2,331
	0,1	
	0,2	
	0,4	
	1,0	
	0,1	
	0,9	
	2,9	z-score > 2
	0,1	
	0,9	
	0,2	
	0,8	outlier (Grubbs)
	0,1	
	0,3	